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Gravimetric Analysis

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GRAVIMETRIC ANALYSIS

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Gravimetric Analysis.

Gravimetric quantitative analysis is that method by which the substance to be estimated is converted into some chemically definite compound, weighed as such, and the amount of the original substance obtained from this weight by calculation. The same definite compound answered both for the estimation of its metal and of its acid. For example, if we precipitated a known weight of silver nitrate with hydrochloric acid we obtained insoluble silver chloride, which was filtered out and weighed, and the amount (x) of Ag in the quantity started with calculated therefrom, because —

Ag Cl : Ag = weight of Ag Cl found : x

If, on the other hand, we started with a known weight of hydrochloric acid, precipitated it with silver nitrate, and collected and weighed the Ag Cl formed, we found the amount (x) of real H Cl actually present in the quantity started with, because —

Ag Cl : H Cl = weight of Ag Cl formed : x

Before giving individual processes for quantitative analysis, we must first say something about the usual manipulation involved.

The Collection and Washing of Precipitates.

When the precipitate has been fully formed and the supernatant fluid has become quite clear, the latter is poured on the filter (which is either previously tared or not, according to circumstances), care being taken not to disturb the precipitate. This was done by holding a glass rod in a perpendicular position over the filter, placing the lip of the beaker against it, and causing the liquid to flow steadily down the rod into the filter. When the latter was three-fourths full, the beaker was turned into an erect position and the rod drained against the inside of the lip, and then laid across the top of the beaker until it was time to refill the filter. After thus pouring off as much as practicable, the precipitate remaining in the beaker was treated with water and well stirred. When the whole had once more settled, the clear fluid was again passed through the filter. This operation having been repeated three or four

times, the precipitate was allowed to pass on to the filter, any particles which adhered to the sides of the beaker having been removed with a rod tipped with a small piece of indiarubber tubing, commonly called a "policeman"; and the whole having been thus collected, the washing was continued by means of a washing-bottle, till the precipitate was quite freed from its soluble impurities. For instance in estimating Aluminium, the aluminium hydroxide was washed till the filtrate no longer gave a turbidity with barium chloride.

Drying of Precipitates.

After the precipitate had been thoroughly washed, the funnel containing it was loosely covered with filter paper and then put into the air-bath till dry. Most precipitates were dried at a temperature of 100° C. (212° F.), but some of them required a heat of 105° C. (220° F.) before becoming constant in weight.

Igniting and Weighing Precipitates.

Many precipitates were first ignited before they could be weighed. This was to drive off the water, which they still retained after drying at 100°C., or to reduce them to a more definite condition. For instance, iron was precipitated as hydrate, but the composition of that body not being constant, it was ignited and so make into pure oxide before weighed. As soon, therefore, as the precipitate appeared dry, it was carefully detached from the filter and put into a previously heated and weighed crucible, the filter was then burnt on the lid (which has been weighed together with the crucible), the ash thrown into the crucible, and the latter covered with the lid. The crucible was supported by a pipe-clay triangle, and gently ignited at first, to prevent spurting from the sudden evolution of steam or gases. The lid was then taken off and the crucible inclined a little, so as to give a free access of air. The ignition was continued for some minutes; and the crucible, having been again covered with the lid, was allowed to cool in a dessicator and weighed.

Some precipitates were not ignited, but weighed on a previously tared filter. Before weighing the filter - for which purpose a weighing

bottle was used - it was dried for about fifteen minutes at 100°C. After drying the precipitate, the filter was again placed in the tube and re-weighed: the increase shown was, of course, the weight of the subatance. It was necessary to replace the filter in the oven for, say, half an hour, and to weigh again. This was repeated until two concordant weights were found.

Analytical Factors for Calculating the Results of Analysis.

To save the working out of a rule-of-three sum on a result of each analysis, it was customary to employ factors. These were obtained by dividing the weight of the required body by the equivalent weight of the body in the form in which it was precipitated. Thus, supposing we were estimating the amount of silver nitrate present in a solution containing .6 gram of the salt, and had precipitated and weighed the same in the form of silver chloride, we would have had

$$\frac{\text{Molecular weight of } AgNO_3}{\text{Equivalent weight of } AgCl} \quad \frac{168.69}{142.3} = 1.1854, \text{ analytical factor.}$$

It then only remained to multiply this factor by the weight of the precipitate to obtain the answer. Let us suppose that the weight of the precipitate was .5 gram, then 1.1854 times .5 = .59270 real $AgNO_3$ present in the .6 gram taken; then $\frac{.59270 \times 100}{.6}$ = 98.79 per cent real $AgNO_3$ present in the sample.

TYPICAL GRAVIMETRIC ESTIMATIONS OF METALS.

Estimation of Aluminium.

This metal was precipitated from the solution of its compounds as aluminium hydroxide by means of ammonia. The hydroxide was then converted by heat into the oxide Al_2O_3, in which form the metal was weighed. This was carried out as follows:-

A stoppered weighing-bottle containing aluminium sulphate was first weighed, after which some of the salt was carefully transferred without loss to a beaker, which was immediately covered with a clock-glass. The bottle was then weighed again, and the difference gave the weight of the

aluminium sulphate taken. The salt was then dissolved in such a quantity of distilled water that the beaker was about one-fourth part filled with liquid and the process of solution aided by gently warming the mixture. A quantity of ammonium chloride solution was then added, equal to about a fourth of the volume of the liquid already present, and then ammonia in the least possible excess beyond the required amount for complete precipitation. The liquid was then boiled until the greater part of the ammonia was expelled, and the issuing steam possessed only a slight smell of the reagent. The precipitate was then collected and washed until entirely freed from all soluble salts, which in this case consisted of sulphates of potassium and ammonium. To ascertain this point, a few drops of the filtrate were collected in a test-tube, and a little barium chloride added. If no precipitate resulted in a few minutes after washing the mixture, the washing could be considered complete. The filter and precipitate were next dried in an air-bath.

While the precipitate was drying, a platinum crucible was heated to redness for a few minutes, cooled in a dessicator, and then weighed. The dried filter containing the aluminium hydroxide was then removed from the funnel, carefully folded up, and placed in the weighed crucible. The crucible, supported upon a clean pipe-clay triangle, was at first very gently heated, the lid having been placed slightly on one side in order to allow the gaseous products of the combustion of the paper to escape, and also to admit the necessary supply of air for the combustion. When the active combustion of the paper had subsided, the temperature was gradually raised and when it had reached a red heat, we removed the lid of the crucible. The vessel was maintained at a bright red heat by means of a good Bunsen flame for about ten minutes. The lid was then replaced, and the crucible transferred to the dessicator to cool, after which it was weighed. It was reheated for a similar time and again weighed, the operation being repeated until two concordant results were obtained.

From the data obtained by the analysis, the percentage of aluminium

was calculated as follows:-

Estimation of Aluminium in Aluminium Sulphate.

Weighing bottle and salt (first weight)	45.9068 gms.
" " " " (second ")	41.9038
Weight of salt taken	4.0030
Weight of crucible and filter ash and Al_2O_3 . . .	11.0906
Weight of crucible alone	10.6598
Weight of Al_2O_3 plus ash	.4308
Weight of ash	.0002
" " Al_2O_3	.4306

Formula weight of Al_2O_3 = 102

" " " Al_2 = 54

Therefore, weight of Aluminium in .4306 gm Al_2O_3 $= \frac{54 \times .4306}{102} = .22796$

hence the percentage of aluminium found = $\frac{0.22796 \times 100}{4.003} = 5.69$ +

Formula weight of $Al_2(SO_4)_3.18\,H_2O$ = 954

Formula weight of Al_2 = 54

hence the theoretical percentage of aluminium in aluminium sulphate $= \frac{54 \times 100}{954} = 5.67$

Estimation of Iron.

The Iron was precipitated from solutions of ferric compounds as ferric hydroxide by means of ammonia. Thehydroxide was converted by heat into ferric oxide, and weighed in that form. The iron in ferrous compounds was first oxidized into the ferric state.

Factor -

(Fe_2O_3) 160 : (Fe_2) 112 = 1: 0.700

Estimation of Iron in Ferric Chloride: The salt was weighed out in a beaker and dissolved in water with the addition of a few drops of hydrochloric acid. The solution was warmed, and, in the case of a ferrous compound, a little strong nitric acid added, sufficient to oxidize the whole of the iron. The mixture was heated nearly to the boiling point for a short time in a covered beaker. Each drop of nitric acid as it was added, produced a brown coloration, owing to the absorption of the disengaged nitric

oxide by the ferrous salt; but when the oxidation was complete, the addition of a drop of the acid produced no visible effect. As a confirmation, the smallest drop of the solution was placed upon a piece of white porcelain by means of a glass rod, and the drop touched with another glass rod which has been dipped into a solution of potassium ferricyanide. If the ferrous iron had been completely oxidized, no blue coloration resulted.

The ferric hydroxide was then precipitated by the addition of a slight excess of ammonia, and the mixture boiled until the steam scarcely smelled of ammonia. The precipitate was decanted with hot water and finally washed upon the filter until the filtrate was entirely free from chlorides. The precipitate was then thoroughly dried in the air-bath. When perfectly dry, as much of the precipitate as possible was detached from the paper, by gently squeezing the cone together, and transferring to a platinum crucible, which was placed on a sheet of white paper. By flattening the paper cone and gently rubbing one side against the other, the remaining adhering particles were detached. The paper was then folded, bound up in a platinum wire, and incinerated as completely as possible. The ash was then shaken off the wire into the crucible, and any particles which might have fallen upon the paper were swept into the crucible. The crucible was then heated to a bright red heat for about ten minutes, after which it was placed to cool in the dessicator and weighed; the heating and weighing having been repeated until no further loss of weight resulted.

Data.

Weight of bottle and salt (first weight)	36.3878	gms.
" " " " " (second ")	35.8728	"
Weight of salt taken	.5150	"
Weight of crucible plus ash plus Fe_2O_3	10.9184	"
" " " alone	10.6614	"
" " Fe_2O_3 plus ash	.2570	"
Weight of ash	.0036	"
" " Fe_2O_3	.2534	"

$.2534 \times .7 = .17738$

$$\frac{.17738 \times 100}{.5150} = 34.44\ \%$$

Estimation of Copper.

The copper was precipitated from solutions of its salts in the form of copper hydroxide by means of Potassium hydroxide. The precipitate was then filtered out, washed and dried. It was then carefully removed from the paper to the weighed crucible, and the filter having been burned on the lid and the ash added to the contents of the crucible, the whole was well ignited, cooled in the dessicator, and weighed rapidly, because cupric oxide is very hygroscopic. To make sure that the oxide contains no cuprous oxide, it wasmoistened with a little fuming nitric acid, dried with the lid on, heated for ten minutes, and then weighed as CuO. This operation required care, being liable to involve loss by spurting.

Estimation of Copper in Copper Sulphate. ($CuSO_4.5\ H_2O$

Weight of bottle plus salt (1st weight)	43.7862
" " " " " (2d ")	42.3718
Weight of salt taken	1.4144
Weight of crucible plus CuO plus ash	11.1162
" " " " ash	10.6624
Weight of CuO plus ash	.4538
Weight of ash	.0036
Weight of CuO	.4502

Amount of Cu in CuO $\ldots\ldots \frac{63.18 \times .4502}{79.18} = .359227$

Amount of Cu in salt $\frac{.359227 \times 100}{1.4144} = 25.39\ \%$

Theoretical amount of Cu in $CuSO_4$ = 25.35 %

Estimation of Silver.

Silver was precipitated from solutions of its salts in the form of silver chloride, Ag Cl, by means of hydrochloric acid, and weighed in this form.

Estimation of Silver in Silver Nitrate: The salt was dissolved in cold water, and the solution acidulated with a little nitric acid. Dilute hydrochloric acid was then gradually added, until complete precipitation had taken place. The mixture was then heated to boiling and the precipitate allowed to settle. The AgCl was then transferred to the filter, and washed with hot water acidified with nitric acid until the filtrate was free from hydrochloric acid (this point was determined by adding silver nitrate to the filtrate). The precipitate was then dried and transferred to a porcelain crucible, which had been previously heated and weighed. Care had to be exercised in folding the paper and binding it with a platinium wire, so that neither the silver chloride nor the recuded silver came in contact with the wire; otherwise the silver would have formed an alloy with the platinum, and vitiated the analysis. The filter ash was deposited in the inverted lid of the crucible, where by means of a pipette, a single drop of nitric acid was allowed to fall upon it, thereby converting the reduced silver into the nitrate. By a similar method a drop of hydrochloric acid was added, which reproduced silver chloride. The lid was then cautiously heated upon a pipe-clay triangle until the acids were completely evaporated. The crucible was then gently heated until the precipitate just began to melt, when it was removed along with the lid to the dessicator, and weighed when cold. It was reheated and again weighed until two concordant weights were found.

Data.

Weight of bottle and salt (1st weight)	36.0210
" " " " " (2d ")	35.2482
	.7728
Weight of AgCl plus crucible plus ash	29.8250
Weight of crucible	29.1680
Weight of AgCl plus ash	.6570
Weight of ash	.0036
Weight of AgCl	.6534

Amount of Ag in AgCl .... $\frac{107.66 \times .6534}{143.1} = .491579$

Amount of Ag in salt ..... $\frac{.491579 \times 100}{.7728} = 63.6\ \%$ *

Theoretical amount of Ag in $AgNO_3$ is 65.45 %.

*This error to a large extent was due to impurities in the salt, as it was prepared by students during the year 1912-13.

Estimation of Lead.

The lead was precipitated as lead sulphate by means of sulphuric acid in the presence of a considerable volume of alcohol.

Estimation of Lead in Lead Acetate, $Pb(C_2H_3O_2)_2 . 3\ H_2O$.

The salt was dissolved in the least amount of water possible and dilute sulphuric acid was added until no further precipitate was produced. Precipitation was made complete by the addition of a quantity of alcohol, and after the precipitate had settled, it was transferred to a filter and washed with methylated spirit (in which lead sulphate is quite insoluble) until the filtrate was free from sulphuric, as was shown by the addition of a few drops of barium chloride to the liquid. The precipitate was then dried and incinerated. The ash, however, after having been moistened with nitric acid, was treated with a single drop of sulphuric acid in order to convert the lead nitrate first formed into lead sulphate. After the excessive acid had been evaporated, the crucible was heated to dull redness, cooled in the dessicator and weighed; the heating, however, had been repeated until the weight was constant.

Data.

Weight of bottle plus salt (1st weight) . . .	36.1194
" " " " " (2d ") . . .	35.3390
Weight of salt	.7804
Weight of $PbSO_4$ plus crucible plus ash . . .	26.8538
" " crucible	26.2256
Weight of $PbSO_4$ plus ash	.6282
" " ash	.0036
" " $PbSO_4$	.6246

Amount of Pb in $PbSO_4$ .... $\frac{207 \times .6246}{303} = .426707$

" " Pb in salt .... $\frac{.426707 \times 100}{.7804} = 54.67\ \%$ *

Theoretical amount of Pb in $Pb(C_2H_3O_2)_2$ = 54.617 %

* This error was also due to a large extent to impurities in the salt, as it was not labelled chemically pure (C. P.)

Estimation of Manganese.

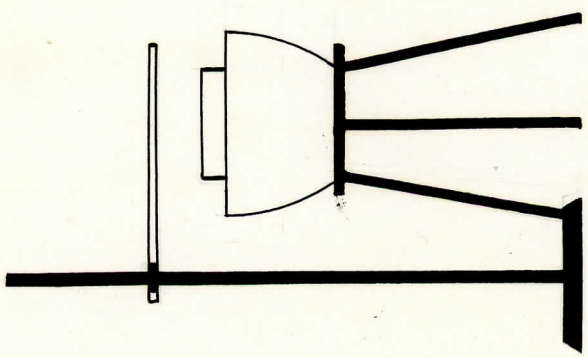
The manganese was precipitated as manganous carbonate by means of sodium carbonate. The carbonate was then converted by heat into the tetroxide, Mn_3O_4 in which form it is weighed.

Estimation of Manganese in Potassium Permanganate.

The powdered salt was weighed out in a beaker and dissolved in water. Suphur doixide was then passed through the solution until the liquid was perfectly clear and colorless. The manganese was next precipitated and washed with boiling water. In this case, however, two special sources of error arose. First, the precipitation of the manganese was not complete, causing, therefore, a loss; and, second, the precipitate carried down with it a certain quantity of the alkali; thus giving rise to a gain. Although by chance these two errors might have neutralized each other, an exact result could only be obtained by adopting the following method for their elimination.

The filtrate, together with the whole of the wash-water, was evaporated to dryness in a porcelain dish. When the liquid reached such a state of concentration that salts began to deposit, the process was finished upon a water-bath. During the entire process care had to be exercised so that no foreign particles fell into the dish; and therefore it was desirable to screen the vessel, by supporting over it a sheet of filter-paper by means of a glass tube bent into a triangle and held by a clamp, as shown in figure.

The residue was then treated with a little hot water, which dissolved the salt, leaving any manganese present in the form of the hydrated oxide. This was filtered through a separate filter, washed with hot water until the wash-water was free from sulphate, and then placed in the air-bath to dry.



The contents of the two filters were then transferred to the platinum crucible, and the two filter-papers incinerated in a coil of platinum wire, one after the other, and the two ashes deposited in the crucible. The crucible was then raised to a bright-red heat for about ten minutes; then cooled in the dessicator.

The residue in the crucible was then treated with a little boiling water, the particles having been reduced to a powder by means of a short piece of glass-rod with smooth ends. The water was poured off through a filter, and the residue washed by decantation three or four times. It was then entirely transferred to the filter, and thoroughly washed with hot water until the washings ceased to give a yellow coloration to the flame when heated upon a loop of clean platinum wire. The residue was next dried in the air-bath and afterwards transferred to the crucible; the filter was incinerated, and the crucible again strongly heated and allowed to cool in the dessicator, and weighed. The heating, however, was repeated until the weight remained constant.

Data.

Weight of bottle plus salt (1st weight)	36.4860
" " " " " (2d ") . . .	35.6788
Weight of salt	.8072
Weight of Mn_3O_4 plus crucible plus ash	11.0628
Weight of crucible	10.6616
Weight of Mn_3O_4 plus ash	.4012
Weight of ash	.0108
Weight of Mn_3O_4.	.3904

Amount of Mn_3 in Mn_3O_4..... $\frac{165 \times .3904}{229} = .28129$

Amount of Mn_3 in salt ..... $\frac{.28129 \times 100}{.8072} = 34.84\ \%$

Theoretical amount of Mn in $KMnO_4$ is 34.81 %.

Estimation of Nickel.

The nickel was precipitated as nickelous hydroxide by means of potassium hydroxide. The dried precipitate was converted by heat into Ni O, in which form the metal was weighed.

Estimation of Ni in $NiCl_2 .6 H_2O$. The precipitation was conducted as in the case of copper. The filter was incinerated in a platinum coil, and the precipitate strongly heated in the platinum crucible until the weight was constant. Nickelous hydroxide retains traces of the alkali with some persistance; therefore, when extreme exactness is desired, the precipitate is treated as in the case of manganese.

Data.

Weight of bottle plus salt (1st weight)	35.6256
" " " " " (2d ")	33.9844
Weight of salt	1.6412
Weight of Ni O plus crucible plus ash	11.1640
Weight of crucible	10.6446
" " Ni O plus ash . .	.5194
" " Ash	.0036
" " Ni O	.5158

Amount of Ni in Ni O ... $\frac{58.6 \times .5158}{74.6} = .405172$

" " Ni : Salt ... $\frac{.405172 \times 100}{1.6412} = 24.68\ \%$

Theoretical amount of Ni in Nickel chloride is 24.66 %

Estimation of Potassium.

The potassium was precipitated in the form of the double chloride of potassium and platinum, and the precipitate was weighted without alteration upon a tared filter. This process is only applicable, however, in the case of such potassium compounds as are wholly converted into the chloride by means of hydrochloric acid.

Estimation of Potassium in Potassium Chloride. The salt was weighed out into a small porcelain dish, and dissolved in about 10 cc. of water,

to which two or three drops of hydrochloric acid had been added. A solution of platinum choride was added in slight excess, and the mixture evaporates on a steam-bath until it became semi-solid.

The crystalline residue was rinsed with alcohol, and, after it was allowed to settle, the supernatant liquid was poured off through a previously weighed filter. The precipitate was washed by decantation with alcohol two or three times, and finally transferred to the filter, where it was further washed with alcohol until the filtrate was free from any tinge of yellow color. The filter was then withdrawn from the funnel, carefully folded, and placed between a pair of watch-glasses to dry in the air-bath. It was then heated and weighed until the weight was constant.

Data.

Weight of bottle plus salt (1st weight)	36.3546
" " " " " (2d ")	34.4808
Weight of salt taken	1.8738
Weight of 2 K Cl. $PtCl_4$ plus filter	1.5872
Weight of filter	.9760
Weight of 2 K CL. $PtCl_4$	.6112

Amount of 2K in 2 K Cl.$PtCl_4$ ..... $\frac{.6112 \times 78}{486}$ = .980938

Amount of K in K Cl ..... $\frac{.980938 \times 100}{1.8738}$ = 52.35 %

Theoretical amount of K in K Cl is 52.349 %.

Estimation of Tin.

The tin was precipitated as the sulphide from either "stannous" or "stannic" solutions by means of sulphuretted hydrogen. The dried precipitate was afterwards converted by heating in contact with air, into tin dioxide, in which form it was weighed.

Estimation of Tin in Stannous Chloride. The purified salt was weighed into a beaker and dissolved in a small quantity of dilute hydrochloric acid. The solution was diluted by the addition of about 250 cc. of

moderately warm water, and sulphuretted hydrogen passed through until the liquid was saturated with the gas. The beaker was then gently heated for a short time to a temperature not higher than can be comfortably borne by the hand. This precipitate has a great tendency to pass through the filter, especially during the process of washing. This was overcome by employing a dilute solution of ammonium acetate with a little acetic acid, with which to wash the precipitate. The dried precipitate was transferred to the platinum crucible, and the filter incinerated in a platinum coil. The ash was moistened with a single drop of strong nitric acid, as in the case of copper, and the excess of acid was expelled by a gentle heat. The crucible was then gradually heated, first with its lid on, and afterwards with it removed, until the sulphide was completed converted into the oxide. The last traces of sulphur were expelled by allowing the crucible to partially cool, introducing a few fragments of ammonium carbonate, and again strongly heating. This was repeated until the weight was constant.

Data.

Weight of bottle plus salt (1st weight)	36.2238
" " " " " (2d ")	35.3476
Weight of salt	.8762
Weight of crucible plus SnO_2 plus ash . .	11.2310
" " "	10.6432
Weight of SnO_2 plus ash	.5878
" of ash	.0036
	.5842

Amount of Sn in SnO_2 ... $\frac{118 \times .5842}{150} = .45957$

Amount of Tin in salt ... $\frac{.45957 \times 100}{.8762} = 52.45\ \%$

Theoretical amount of Sn in $SnCl_2.2\ H_2O$ is 52.44 %.

Estimation of Arsenic.

The arsenic was precipitated from solutions of "arsenic" com-

pounds in the form of ammonium magnesium arsenate by means of magnesium sulphate in the presence of ammonia and ammonium chloride. The precipitate was converted by heat into magnesium pyro-arsenate, in which form the metal was weighed.

Estimation of Arsenic in Hydrogen Disodium Arsenate. The salt was weighed out into a wide-mouthed bottle and dissolved in water. A moderate quantity of ammonia was added and then " magnesia mixture" in excess. The mixture was then briskly shaken in the stoppered bottle for about five minutes, after which it was filtered, washed with ammonia and dried.

The dried precipitate was detached from the filter and deposited upon a clock-glass and covered over with a clean beaker. The filter was then moistened with a few drops of an ammonium nitrate solution, and returned to the air-bath. As soon as it was dry, it was incinerated in a Rose's crucible. The precipitate was then transferred to it, after which it was heated in a gentle stream of oxygen. The heat, which was applied very gradually at first, was finally increased to a bright redness. After it was cooled and weighed, it was reheated in oxygen until the weight was constant.

Data.

Weight of bottle plus salt (1st weight)	37.0824
" " " " " (2d ")	35.6482
Weight of salt . . .	1.4342
Weight of crucible plus $Mg_2As_2O_7$ plus ash	29.1130
Weight of crucible	28.5560
Weight of $Mg_2As_2O_7$ plus ash	.5570
Weight of ash . . .	.0036
Weight of $Mg_2As_2O_7$	.5534

Amount of As_2 in $Mg_2As_2O_7$ ..... $\frac{150 \times .5534}{310} = .267774$

" of Arsenic in salt .... $\frac{.267774 \times 100}{1.4342} = 18.66\ \%$.

Theoretical amount of Arsenic in Hydrogen Disodium Arsenate is 18.65%.

Separation of Silver and Copper.

The metal was dissolved in nitric acid, and the silver precipitated as silver chloride. This was separated and weighed as such. The copper was precipated from the filtrate in the form of copper hydroxide, which was converted by heat into copper oxide, and weighed in this form.

Analysis of Silver Coinage. The silver coin (a ten cent piece) was carefully weighed, and then dissolved in a little nitric acid in an Erlen-meyer flask. The acid, consisting of strong nitric acid, to which about half its volume of water had been added, was introduced by means of a small funnel, which was allowed to remain in the mouth of the flask during the whole process, as it prevented loss of the liquid by spurting. The solution was started by the application of heat. When the metal was entirely dissolved, the excess of acid was evaporated off by gently boiling the solution. The liquid was then diluted with water and transferred to a beaker, the flask and funnel having been thoroughly rinsed once or twice with distilled water, so that the whole of the solution was transferred without loss. From this solution the silver chloride was precipitated, and the silver estimated as described in an earlier part of this paper. The filtrate, together with the washings containing the copper, was heated to boiling and determined according to previous description.

Data.

Weight of coin 2.4852

" " crucible plus Ag Cl plus ash . . . 29.4800

Weight of crucible 29.1790

" " Ag Cl plus ash 3010

(Bro't. forward, Weight of Ag Cl-ash) .3010

Weight of ash 0036

Weight of Ag Cl 2974

.2974 x .75202 * = .223651

Amount of Ag in coin ..... $\frac{.223651 \times 100}{2.4852}$ = 89.99 %.

Weight of crucible plus Cu O plus ash 29.4944

Weight of crucible 29.1786

" of Cu O plus ash .3158

" of ash 0036

" of Cu O 3122

.3122 x .79823 * = .249207

Amount of Cu in coin ..... $\frac{.249207 \times 100}{2.4852}$ = 10.03 %.

* Analytical factors.

The above are in short the operations carried out in the Chemical Laboratory of Ursinus College during the year 1912-13 under the direction of Dr. Matthew Beardwood.